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The Thermal Deprotection Process in an E-beam Exposed,
Phenolic-Based Polymer

by

Treva Long, S. Kay Obendorf, and Ferdinand Rodriguez

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An alkali soluble latent image can be generated in pure PBOCST (poly(tert-butoxycarbonoxystyrene)) by e-beam exposure followed by a post-exposure bake (PEB) at relatively high temperatures (140°-150°C). Pattern wet development operates on the same basis as a PBOCST/acid generator two component system. Deprotection (heating), accelerated by the exposure, yields alkali soluble areas while unexposed areas remain insoluble. Sensitivities of 5-10 $\mu C/cm^2$ and contrasts of 6-10 have been measured. Complete TBOC deprotection results in a film thickness loss of 35-40% as CO_2 and isobutene are evolved. A laser interferometer/hotplate setup was used to track film thickness with time during the thermal deprotection process. IR analysis confirmed the correlation between conversion and thickness. It was found that a slow initial conversion rate was followed by an exponentially steep rise in rate after the 50% conversion point. Activation energies were on the order of 30 kcal/mol. It is apparent that thermolysis in both exposed and unexposed resist occurs during the PEB but that the difference in extent of conversion on exposure is sufficient to generate a pattern.

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The Thermal Deprotection Process in an E-beam Exposed, Phenolic-Based Polymer

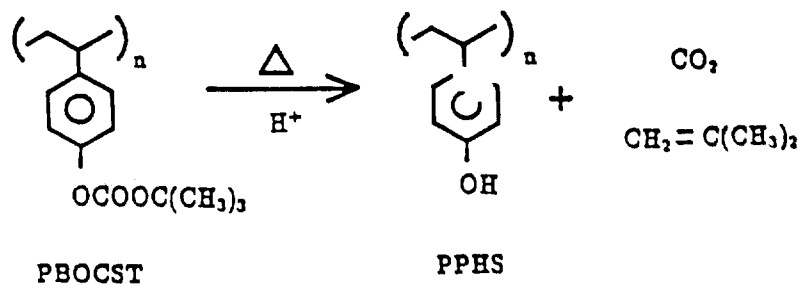Treva Long[1], S. Kay Obendorf[1], and Ferdinand Rodriguez[2],
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Abstract

In our current work, we have examined the e-beam sensitivity and thermal processing behavior of pure PBOCST (poly(tert-butoxycarbonoxystyrene)). An alkali soluble latent image can be generated in the PBOCST by e-beam exposure followed by a post-exposure bake (PEB) at relatively high temperatures (140-150°C). Pattern wet development operates on the same basis as a PBOCST/acid generator two component system. Deprotection, accelerated by the exposure, yields alkali soluble areas while unexposed areas remain insoluble. Sensitivities of 5-10 $\mu C/cm^2$ and contrasts of 6-10 have been measured. Complete TBOC deprotection results in a film thickness loss of 35-40% as CO_2 and isobutene are evolved. A laser interferometer/hotplate setup was used to track film thickness with time during the thermal deprotection process. IR analysis confirmed the correlation between conversion and thickness. It was found that a slow initial conversion rate was followed by an exponentially steep rise in rate after the 50% conversion point. Activation energies were on the order of 30 kcal/mol. It was concluded that thermolysis in both exposed and unexposed resist occurred during the PEB but that the difference in extent of conversion on exposure was sufficient to generate a pattern.

1 Introduction

Wafer throughput in microlithography is dependent on the radiation sensitivity of the resist film. The lower the dose needed to produce a latent image in the resist, the higher the throughput. One route to higher sensitivity is through chemical amplification (CAMP). A single radiation event generates a moiety which catalyzes numerous reactions before being consumed. One of the most successful CAMP resists is PBOCST, the tert- butoxycarbonate (tBOC) protected form of poly(para- hydroxystyrene) (PPHS). When in the tBOC protected form, the polymer is only soluble in nonpolar solvents. Removal of the tBOC group by acidolysis or thermolysis yields PPHS, an alkali soluble polymer.

$$\left(\begin{array}{c}\curlywedge\end{array}\right)_n \quad \xrightarrow[H^+]{\triangle} \quad \left(\begin{array}{c}\curlywedge\end{array}\right)_n \quad + \quad CO_2$$

$$OCOOC(CH_3)_3 \qquad\qquad OH \qquad\qquad CH_2 = C(CH_3)_2$$

$$PBOCST \qquad\qquad\qquad PPHS$$

In microlithography applications, an acid generating species (AG) is blended with the PBOCST. Upon exposure, the AG produces a strong acid. A short post-exposure bake (PEB) step at low temperatures activates the acid in the exposed areas to convert the PBOCST to PPHS leaving unexposed areas uneffected [1]. The latent image is then wet-developed using aqueous alkali. Two major advantages of the PBOCST/AG resist are elimination of toxic organic solvents in development and absence of swelling during development. Swelling does not occur since the alkali selectively wet-etches the hydrophilic PPHS without interacting with the hydrophobic PBOCST.

The two component PBOCST/AG system has been shown to produce submicron images using deep UV (DUV), e-beam or X-rays. Umbach, et. al. [2] used PBOCST with an unspecified acid catalyst to fabricate structures with 18 nm linewidths at an e-beam dose of 50 $\mu C/cm^2$. Ito, et. al. [3] have evaluated PBOCST sensitized with 5-15 wt% onium salts as a negative-working resist. E-beam exposed films were post-baked at 100 or 130°C for 2 min and developed with anisole. Sensitivities of 1-3 $\mu C/cm^2$ and contrasts of 2.3 were reported. DUV sensitivities are typically 20 mJ/cm^2 or less, again with PEB temperatures around 100°C [4].

Onium salts, the common AG component of the PBOCST system, are hard to synthesize and often toxic. Less efficient acid generators, such as the nitrobenzyl tosylate compounds, have been tested as onium salt substitutes. Unfortunately they can reduce the sensitivity of the CAMP system by almost an order of magnitude [4]. A more radiation sensitive CAMP polymer can compensate for this reduced sensitivity. Copolymers of PBOCST and SO_2 (poly(tBOChydroxystyrene-sulfur dioxide), TBSS) incorporate a C-S bond into the backbone that will easily scission upon exposure [5]. In a positive-working scheme, the acid catalyzed deprotection reaction acts together with chain scissioning to yield a resist with increased alkali solubility in the exposed area. TBSS/tosylate DUV sensitivities are in the range of 50 mJ/cm^2 while the contrast achieved with TBSS/tosylate exceeds that of the original TBOCST/onium salt [4]. Two-component systems can be more complex to characterize and control than simple one component resists. Novembre, et. al. [6] have shown that TBSS alone can function as a chemically amplified X-ray or electron beam resist. Its sensitivity depends on the TBOCST-sulfur dioxide ratio. For a 2:1 TBSS:SO2 copolymer, a dose of 90 $\mu C/cm^2$ resolved 0.25 μm l/s patterns [7]. Radicals produced by the exposure were proposed as being responsible for acid formation and the CAMP-like performance. As with the two component systems, heat treatment after exposure was critical in developing the latent image.

This work investigates the thermal deprotection behavior of PBOCST films as monitored with a laser interferometer/hotplate apparatus. Deprotection of PBOCST films results in a significant loss of thickness as volatile products are evolved. Laser interferometry is an effective method for tracking thickness losses and hence for measuring the deprotection rate under various conditions. We have found that pure PBOCST has potential as an electron-beam sensitive, CAMP-like resist when coupled with the proper thermal processing conditions. Thermal deprotection approximates autocatalytic behavior with the reaction half-life dependent on electron-beam dose. Differences in extent of conversion between exposed and unexposed regions are sufficient to yield submicron features in the PBOCST resist.

2 Experimental

2.1 PBOCST Synthesis

Three Hoechst Celanese PPHS's, manufacturer's MWs of 6500, 17500 and 56200, were used as the starting materials. Hoechst Celanese produced these by suspension polymerization of acetoxystyrene monomer and subsequent hydrolysis of the acetoxy group to the hydroxy group. Conversion of the hydroxy group to the tBOC group to obtain PBOCST proceeded via the method described by Houlihan, *et. al.* [8]. IR spectra confirmed that the conversion of the hydroxy groups was complete.

2.2 GPC Evaluation

Molecular size determinations of the PBOCST polymers were made using a Waters gel permeation chromatograph (GPC) system equipped with duPont Zorbax columns. Tetrahydrofuran was used as the eluting solvent in all cases. Polystyrene calibration standards were used for MW correlations. The PBOCST MW distributions are listed in Table 1. The molecular weights reported by the manufacturer were obtained by GPC of the acetoxy precursor polymers followed by adjustment for the change in mass on hydrolysis to PPHS. Conversion of PPHS to PBOCST should increase M_w only by a factor of 1.83 (220/120). The fact that M_w is larger than predicted for the PBOCST may be due to a difference in interaction of the polymer with column packing *vis a vis* the acetoxy precursors.

2.3 Laser Interferometer and Thermal Processing

Thermal processing behavior of PBOCST (6500 MW) was evaluated using a laser interferometer/hotplate setup. Films of approximately 1.5 μm thickness were spin-cast from chlorobenzene onto 3" diameter silicon wafers and prebaked at 105°C for 1/2 hr. A beam from an unpolarized He-Ne laser (2 mW, 632.8 nm) was reflected at an angle of about 10° from the surface of a coated wafer placed on the hotplate. The reflected light beam was collected by a photocell coupled through an amplifier to a chart recorder. An aluminum hotplate equipped with an Omega programmable temperature controller provided uniform temperatures at the wafer's surface to within ± 2°C . Input to the temperature controller was from a surface thermocouple bonded to the top of the coated Si wafer. Rates of conversion were measured from 130 to 155°C .

Conversion of PBOCST films to PPHS results in a 35-40% thickness decrease [10] as volatile products are lost. As the film thickness changes, thin film interference effects cause a sinusoidal oscillation in intensity. Each oscillation or fringe can be translated into an equivalent thickness loss, d_p [9], using:

$$d_p = \frac{\lambda}{2(n_2^2 - n_1^2 \sin^2 \alpha)^{1/2}} \tag{1}$$

$$\lambda = \text{light wavelength}$$
$$n_2 = \text{polymer refractive index}$$
$$n_1 = \text{air refractive index}$$
$$\alpha = \text{incident angle}$$

Analysis of interferometer data for a film with time-varying composition must take into account the changing refractive index in eqn 1. Refractive indices of PBOCST and PPHS

are 1.53 and 1.61, respectively, as measured by ellipsometry. Given that d_p will differ by only 5% for these polymers, the interferometer traces were interpreted based on an average of 203 nm/fringe. The effect of electron beam exposure on the thermal processing behavior was also tested on the interferometer/hotplate by flood exposing PBOCST films at 20 μC/cm^2 prior to the bake step. Films were exposed to a wide beam of electrons with a modified JEOL JEM7A transmission electron microscope (50 kV). Incident electron doses were determined from the exposure time and the electron charge density, which was measured with a Faraday cup.

2.4 IR Evaluation

To correlate the film thickness during thermolysis with composition, PBOCST films at various bake stages were analyzed by IR. Films were spun onto Si wafers from chlorobenzene and baked at 140 or 150°C at times ranging from 0 to 50 mins. IR spectra for these samples were recorded on a Perkin Elmer 683 infrared spectrophotometer. Extent of conversion as reflected by the carbonyl peak intensity at 1755 cm^{-1} agreed well with fractional conversion based on film thickness changes.

2.5 Lithographic Evaluation

Films for lithographic evaluation were spin-cast at 1500 rpm from 15-25 wt% solutions in chlorobenzene onto 3" silicon wafers. The films were prebaked at 105°C for 30 min. Film thicknesses were 1300, 1500 and 1100 nm for polymers A, B and C (Table 1) respectively, as measured with a Leitz Film Thickness Interferometer. Test patterns (for sensitometry and evaluation of contrast) were exposed using a Cambridge EBMF 10.5 at Cornell's National Nanofabrication Facility (NNF). All patterns were exposed using an accelerating voltage of 20 kV. The exposed patterns were post-baked for 38-39 min at 150°C in a forced air oven. Patterns were dip-developed in Shipley MF319 (a TMAOH aqueous base formulation) at 23°C , followed by a deionized water rinse. Resolution of the patterns was evaluated using a Cambridge S200 SEM.

3 Results and Discussion

3.1 Laser Interferometer/Hotplate

Figure 1 is an actual interferometer trace for the conversion of a PBOCST film to PPHS at 140°C . Over a 51 min bake time approximately 3.5 fringes or an equivalent film loss of 710 nm occurs. The conversion is initially slow but accelerates rapidly after 40 mins such that the bulk of the film loss is in the last 10 min bake segment. All interferometer traces for bake temperatures of 130 through 155°C and for unexposed and exposed films follow this same pattern.

The interferometer data can be translated into fractional film thickness loss vs time by defining
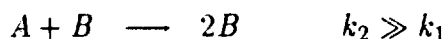
$$f \ = \ \frac{\Delta d}{\Delta d_{total}} \tag{2}$$

where Δd is the thickness loss at time t and Δd_{total} is the total thickness loss observed. Plots of f vs time for bake temperatures of 140 and 150°C are sigmoidal but with very different

time scales (Figure 2). Included in Figure 2 for comparison with the interferometer data is the IR analysis of the PBOCST films. The absorbance of the C=O peak at 1755 cm⁻1 was chosen as a measure of the extent of deprotection. Good agreement between IR and interferometer data allows the fractional film thickness loss to be interpreted as an extent of conversion for the reaction.

The half-life $t_{1/2}$ of a reactant is defined as the time at which its concentration is halfway between its initial and final values. For the PBOCST films $t_{1/2}$ is the time corresponding to $f = 1/2$. If f is plotted against a reduced time $\theta = t/t_{1/2}$, the interferometer curves all collapse onto one master curve regardless of bake temperature or e-beam exposure dose (see Figure 3). Table 2 lists $t_{1/2}$ measured for interferometer runs at temperatures from 130 to 155°C and for e-beam exposures of 0 and 20 μC/cm². As an approximation, the half-life is inversely related to the reaction rate constant. Plotting $t_{1/2}$ vs 1000/T (Figure 4) yields straight lines with a slope of Ea/R where Ea is an activation energy. Both unexposed and exposed films have activation energies of 31 kcal/mol. The constant offset between the lines indicates that the half-life of the unexposed films is approximately 20% higher than the half-life of the exposed films.

Ito, et. al. [11] have shown that the thermolysis of PBOCST is autocatalytic: a small amount of phenol, a weak acid, present in the PBOCST is enough to catalyze the tBOC loss. Phenol groups thus formed by an initially slow thermolysis could catalyze tBOC conversion at a much more rapid pace during the later stages of the film deprotection. The standard model for autoaccelerated reactions is based on two reactions taking place [12]. The first is a very slow residual reaction of the reactant (A) to a product (B). The second is a very fast reaction of A to B catalyzed by the presence of B.

$$A \longrightarrow B \qquad k_1 \text{ small}$$

$$A + B \longrightarrow 2B \qquad k_2 \gg k_1$$

The net result is that the conversion, f, goes from 0 to 1 in terms of a reduced time $\theta = t/t_{1/2}$ according to

$$\frac{f}{1-f} = \frac{r}{1-r}(r^{-\theta} - 1) \tag{3}$$

where $r = k_1/k_2 A_o \ll 1$ and A_o is the initial concentration of A. In Figure 3 is a plot of Eqn 3 for $r = 0.001$. The shape is sigmoidal but does not rise as steeply after $f = 0.5$ as does the PBOCST conversion data plotted with it. The fact that the measured data diverges from the model at high conversion is understandable since the system is a solid undergoing a change in mass rather than an idealized solution. A rough fit of the experimental data from $f = 0$ to 0.5 using equation 3 yields activation energies of 46 and 26 kcal/mol for rate constants k_1 and k_2 respectively. The relative values of these activation energies is reasonable. The slow initial conversion of A to B is highly temperature dependent while the autocatalyzed reaction is less temperature sensitive.

3.2 Patterning characteristics

Exposure to radiation followed by an appropriate PEB makes exposed portions of the film soluble in base and leaves the unexposed areas insoluble. PBOCST films were line-exposed

at dosages of 4-10 $\mu C/cm^2$ and postbaked at 150°C for 36-39 min. Increasing or decreasing the bake time by as little as 3-4 min yielded over- or under-developed line profiles. Dip-development of the patterns for 10-20 sec in MF319 gave good line profiles with minimal lift-off problems. Development using KOH or NaOH solutions produced severe undercutting and lift-off. Figure 5 shows photos of 1 μm features for each resist MW. The profile photomicrographs show that the resist features are significantly undercut with the PBOCST A but not with higher molecular weights. Figure 6 shows photos of 0.5 μm line features for B. Very little swelling was observed during development as one might expect when the differential dissolution is controlled by a change in polymer polarity.

3.3 Sensitivity and Contrast

Resist performance is reflected by sensitivity and contrast. Determining contrast requires measurement of the thickness of the resist remaining as function of exposure dose. The contrast, γ, is defined as

$$\gamma = \left[\log \frac{D_1}{D_0}\right]^{-1} \tag{4}$$

where D_0 and D_1, the dose at which the dissolution begins and the dose at complete development, are determined by drawing a tangent line through the inflection point of the exposure curve. Gamma curves for the PBOCST resists are shown in Figure 7. Contrast values of 10, 4 and 6 were computed for A, B and C respectively. In addition, the sensitivity (electron dose at which half the film thickness remains) ranged from a low of 4.8 $\mu C/cm^2$ for C to 8.3 $\mu C/cm^2$ for B.

3.4 Lithographic Mechanism

Image formation in PBOCST is controlled by the extent of conversion of the tBOC group to the hydroxy functionality. Two component PBOCST/AG systems operate on the basis of an acid-catalyzed deprotection of tBOC in the exposed areas while unexposed areas remain in the PHOST form. Exposure to radiation produces an acid from the acid-generating component. Low PEB temperatures then yield complete deprotection within a few minutes. In the pure PBOCST system the mechanism for producing the latent image is less apparent. Changes in the film induced by the electron-beam exposure require higher PEB temperatures for long times to produce a developable image. Thermolysis rather than acidolysis of the tBOC groups predominates in the deprotection process. The electron-beam induced change between the exposed and unexposed film is magnified during this thermolysis. Results from the laser interferometer emphasize the non-linear nature of the deprotection reaction in the pure PBOCST system. A slow initial conversion stage is followed by a period of extremely rapid conversion. If even only a small quantity of acidic phenols are generated in the exposed film, those regions may reach and go through the second phase of rapid converision while the unexposed regions lags behind in the slow conversion phase. Stopping the thermal treatment before unexposed regions can achieve high conversion levels leaves a sufficient percentage of nonpolar tBOC groups to prevent dissolution in alkali. At what extent of tBOC conversion does the polymer film become base soluble? Nalamasu et. al. [5] have noted an almost step-function-like dependence of TBSS alkali development on the tBOC concentration. When tBOC content is less than 5% the film becomes alkali

soluble. Laser interferometer results indicate that tBOC conversion of unexposed areas is only from 40 to 80% complete when the 20 $\mu C/cm^2$ areas have achieved 100% conversion.

4 Conclusions

When an acid generator is present in a CAMP resist formulation, the mechanism for producing a lithographic pattern is straightforward. The strong acid generated causes deprotection at a relatively low temperature with an activation energy of about 11 to 14 kacl/mol. We have found that PBOCST alone can function as a CAMP e-beam resist. The e-beam exposure may cause the production of acidic phenolic groups in the film resulting in an autoaccelerated deprotection of exposed regions during the PEB. Thermolysis in both exposed and unexposed resist occurs in the PEB but the difference in extent of conversion is sufficient to generate a pattern that can be developed with aqueous alkali. The energy of activation (31 kcal/mol) is much higher for this autocatalyzed reaction than for the strong acid case.

Acknowledgements

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Table 1: Molecular weight (polystyrene equivalent) of PBOCST

Sample	M_n	M_w	Polydispersity	Manufacturer's M_w
A	4900	10000	2.0	6500
B	17900	62000	3.5	17500
C	57800	168000	2.9	56200

Table 2: Half-life PBOCST films at exposures of 0 and 20 μC/cm^2

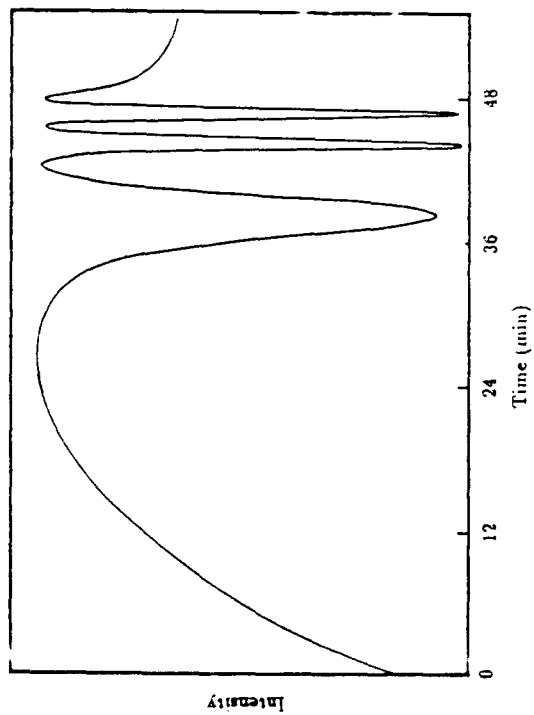| Temperature (°C)	Half-life for 0 μC/cm^2 (min)	Half-life for 20 μC/cm^2 (min)
130	109.2	97.6
135	68.8	58.1
140	43.0	36.2
145	26.8	22.8
150	16.7	14.1
155	11.8	10.1

Figure 2 Fraction conversion vs bake time for 140°C and 150°C. Solid lines represent interferometer data and filled and open circles represent infrared data.
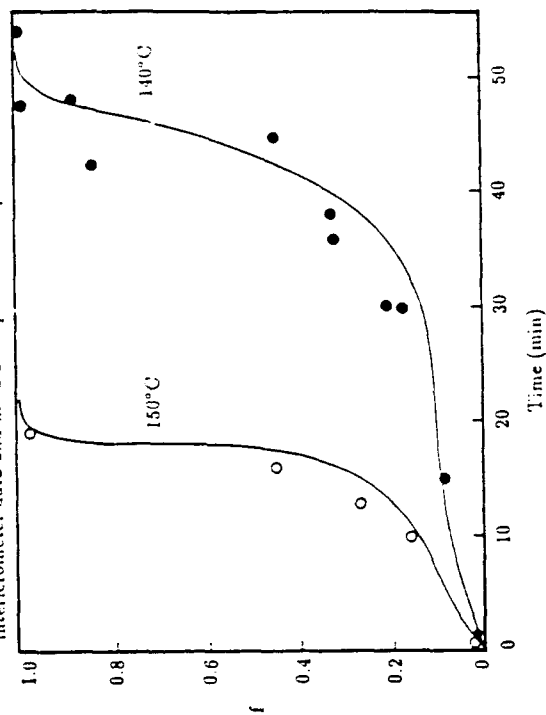

Figure 4 Half life vs 1000/T for deprotection of PBOCST film.

Figure 1 Laser interferometer trace for PBOCST film baked at 140°C on hotplate.

Figure 3 Fraction conversion vs reduced bake time. Dashed line represents autocatalytic model (Eqn 3).

Figure 7   Lithographic response of Polymers A, B and C.

Polymer A
Polymer B
Polymer C

PEB 150°C in air oven

Developer Shipley MF319

Normalized film thickness

Incident E-beam Dose ($\mu C/cm^2$)

Figure 5   1 μm line features in Polymer A. Exposure dose 8 $\mu C/cm^2$.

Figure 6   0.5 μm line features in Polymer B. Exposure dose 29 $\mu C/cm^2$.

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